P / IB 99/01747
13. 12. 99

P 023

REC'D 14 DEC 1999
WIPO PCT

Sertifikaat

REPUBLIEK VAN SUID-AFRIKA

Certificate

IB 99/1747

4

PATENTKANTOOR

PATENT OFFICE

DEPARTEMENT VAN HANDEL EN NYWERHEID

REPUBLIC OF SOUTH AFRICA

DEPARTMENT OF TRADE AND INDUSTRY

Hiermee word gesertifiseer dat
This is to certify that the attached documents attached hereto are true copies of the Forms P2, P6, provisional specification and drawings of South African Patent Application No. 98/9866 in the name of BRITS, Willem Hendrik (assigned from IMMALYTICA (PROPRIETARY) LIMITED, formerly ISLANDSITE INVESTMENTS ONE (PTY) LTD)

Filed	:	29.10.98
Entitled	:	ASSAYING

PRIORITY DOCUMENT

SUBMITTED OR TRANSMITTED IN COMPLIANCE WITH RULE 17.1(a) OR (b)

Geteken te / Signed at PRETORIA in die Republiek van Suid-Afrika, hierdie / in the Republic of South Africa, this 12th dag van / day of November 1999

Registrateur van Patente
Registrar of Patents

FORM P.2

REPUBLIC OF SOUTH AFRICA	REGISTER OF PATENTS	PATENTS ACT, 1

OFFICIAL APPLICATION			LODGING DATE: PROVISIONAL		ACCEPTANCE DATE	
21	01	989866	22	29 OCTOBER 1998	47	
INTERNATIONAL CLASSIFICATION			LODGING DATE: COMPLETE		GRANTED DATE	
51			23			

FULL NAME(S) OF APPLICANT(S)/PATENTEE(S)

71 ISLANDSITE INVESTMENTS ONE (PTY) LTD

Name changed: Immalytica (Pty) Ltd.
27-1-99

AANSOEKERS VERVANG
APPLICANTS SUBSTITUTED

APPLICANTS SUBSTITUTED		DATE REGISTERED
71	Willem Hendrik Brits	22-10-99

ASSIGNEE(S)		DATE REGISTERED
71		

FULL NAME(S) OF INVENTOR(S)

72 WILLEM HENDRIK BRITS

PRIORITY CLAIMED	COUNTRY		NUMBER		DATE	
N.B. Use International abbreviation for country (see Schedule 4)	33	NONE	31	NONE	32	NONE

TITLE OF INVENTION

54 ASSAYING

ADDRESS OF APPLICANT(S)/PATENTEE(S)

29 BOEKENHOUT STREET, DELPARK, EXT 5, BRAKPAN, SOUTH AFRICA

ADDRESS FOR SERVICE		S AND F REF
74	SPOOR AND FISHER, SANDTON	JP/I 174/DC/acm

PATENT OF ADDITION NO.		DATE OF ANY CHANGE
61		

FRESH APPLICATION BASED ON	DATE OF ANY CHANGE

SPOOR AND FISHER

REPUBLIC OF SOUTH AFRICA
PATENTS ACT, 1978
APPLICATION FOR A PATENT
AND ACKNOWLEDGEMENT OF RECEIPT
(Section 30 (1) – Regulation 22)

REPUBLIC OF SOUTH AFRICA FORM P.1
REVENUE
29.10.98 R 060,00
MASB 505
INKOMSTE
REPUBLIEK VAN SUID AFRIKA

The granting of a patent is hereby requested by the undermentioned applicant on the basis of the present application filed in duplicate

OFFICIAL APPLICATION NO.

21	01	989866

S AND F REFERENCE

JP/I 174/DC/acm

FULL NAME(S) OF APPLICANT(S)

71	~~ISLANDSITE INVESTMENTS ONE (PTY) LTD~~ Name changed 27-1-99 ~~Immalytica Pty Ltd~~ Willem Hendrik Botes 22-10-99

AANSOEKER VERVANG / APPLICANT SUBSTITUTED

ADDRESS(ES) OF APPLICANT(S)

	29 BOEKENHOUT STREET, DELPARK, EXT 5, BRAKPAN, SOUTH AFRICA

TITLE OF INVENTION

54	ASSAYING

THE APPLICANT CLAIMS PRIORITY AS SET OUT ON THE ACCOMPANYING FORM P.2 THE EARLIEST PRIORITY CLAIM IS:

COUNTRY: NONE NUMBER: NONE DATE: NONE

~~THIS APPLICATION IS FOR A PATENT OF ADDITION TO PATENT APPLICATION NO.~~

21	01	

~~THIS APPLICATION IS A FRESH APPLICATION IN TERMS OF SECTION 37 AND IS BASED ON APPLICATION NO.~~

21	01	

THIS APPLICATION IS ACCOMPANIED BY:

- ☒ 1. A single copy of a provisional or two copies of a complete specification of 13 pages.
- ☒ 2. Drawings of 2 sheets.
- ☐ 3. Publication particulars and abstract (Form P.8 in duplicate).
- ☐ 4. A copy of Figure of the drawings (if any) for the abstract.
- ☐ 5. An assignment of invention.
- ☐ 6. Certified priority document(s) .
- ☐ 7. Translation of the priority document(s).
- ☐ 8. An assignment of priority rights.
- ☐ 9. A copy of the Form P.2 and the specification of S.A. Patent Application No.
- ☐ 10. A declaration and power of attorney on Form P.3.
- ☐ 11. Request for ante-dating on Form P.4.
- ☐ 12. Request for classification on Form P.9.
- ☒ 13. Form P.2 in duplicate.

74	ADDRESS FOR SERVICE: SPOOR AND FISHER, SANDTON

Dated: 29 OCTOBER 1998

REGISTRAR OF PATENTS, DESIGNS, TRADE MARKS AND COPYRIGHT
1998 -10- 29
PRETORIA
REGISTRATEUR VAN PATENTE, MODELLE, HANDELSMERKE EN OUTEURSREG

REGISTRAR OF PATENTS

SPOOR AND FISHER
PATENT ATTORNEYS FOR THE APPLICANT(S)

SPOOR AND FISHER

FORM P.6

REPUBLIC OF SOUTH AFRICA
PATENTS ACT, 1978

PROVISIONAL SPECIFICATION

(Section 30(1) – Regulation 27)

OFFICIAL APPLICATION NO.			LODGING DATE	
21	01	989866	22	29 OCTOBER 1998

FULL NAME(S) OF APPLICANT(S)

71	ISLANDSITE INVESTMENTS ONE (PTY) LTD Name changed 27-1-99 Immalytica (Pty) Ltd

[illegible] ERVANG Willem Hendrik Brits 22-10-99
A [illegible] SUBSTITUTED

FULL NAME(S) OF INVENTOR(S)

72	WILLEM HENDRIK BRITS

TITLE OF INVENTION

54	ASSAYING

BACKGROUND OF THE INVENTION

This invention relates to assaying.

In order to plan and manage mining operations and to estimate recoverable ore reserves it is necessary to have the facility to determine the concentration in ore samples of precious metals, typically gold and PGMs (platinum group metals

including platinum, palladium, rhodium, osmium, indium and ruthenium). It is already known to use a fire assay process for this purpose. In fire assay, PGMs and gold are separated from gangue materials by collection into either lead or nickel sulphide at temperatures of around 1200 - 1450°C. This is achieved by mixing an aliquot of the sample with a flux containing either lead oxide, for the lead collection, or a combination of nickel carbonate and sulphur, for the nickel sulphide, with other chemicals. This mixture is placed into a ceramic crucible, which in turn is placed into an electric or gas fired furnace and heated to an appropriate temperature for a period of about 90 minutes. During this time the mixture melts and, because their chemical affinity, PGMs and gold are collected into either lead or nickel sulphide. On cooling the lead or nickel sulphide is separated from the gangue material and the PGMs and gold content determined by a number of analytical techniques.

The advantage of Fire Assay collection over other analytical procedures, is that it effectively concentrates PGMs and gold from a large sample aliquot into a media which is much more amenable to further treatment for the separation and analysis of the PGMs and gold. Fire Assay is, therefore, used extensively in all laboratories engaged in the analysis of samples containing precious metals and, indeed, is an essential stage in the analysis of lower grade samples such as concentrates, feed and tails.

In electrically heated furnaces, radiant heat from resistive elements is applied to the prepared ore samples. It is difficult to maintain constant operating conditions in an electrical furnace. Furthermore, resistive elements are fragile and deteriorate over time. This variability in furnace conditions detrimentally affects the accuracy of assay results and gives rise to excessive repeat rates.

Gas fired furnaces, on the other hand, are noisy and require frequent maintenance, which lengthens the average time taken to complete an assay.

The flux used in assaying contains lead and is environmentally hazardous, either in dust form during the flux preparation stage, or as fumes which form during the fusion and cuppelation stages of the process. Personnel involved in the assaying process require monitoring of lead blood levels every six months, which is expensive and disconcerting to the persons involved.

The fusion pots used for fusion of the flux / ore sample combinations are brittle and break easily. In order to overcome this difficulty, each assay is duplicated or triplicated, to ensure that at least one successful assay result is obtained, which increases the overall assaying costs. Further, the duplicated or triplicated samples are fused in different furnaces in order to compensate for the variability in furnace conditions.

The recovery of lead from the fusion slag is also hazardous as the slag is broken up, usually manually, by impact to liberate globules of lead entrapped in the slag. Slivers of slag are sharp and necessitate the wearing of adequate safety equipment. Generally, not all lead globules are usually liberated from the slag, leading to an inevitable loss of lead.

Current assay techniques are labour intensive and, therefore, prone to human error. The average time taken to complete an assay normally exceeds twelve hours. It is desired to improve the accuracy, the turnaround time and the safety aspects of known prior art assaying techniques.

SUMMARY OF INVENTION

According to the invention there is provided a method for assaying an ore sample to determine the concentration of selected metals therein, comprising the steps of:

preparing an ore sample;

combining the prepared ore sample with a lead-containing flux;

inductively heating the combination at a predetermined reference temperature profile to form a fusion of slag and lead containing the metals in the sample;

separating the lead from the slag; and

determining the concentration of the selected metals in the sample.

The sample of ore and flux is preferably mixed in a container made from carbon-based material, and the container is inductively heated with the sample and flux.

Preferably, the container is made from plastics material and comprises a lid which is arranged to close the container sealingly.

The container may also comprise identification means in the form of a bar code for identifying the sample contained therein.

Advantageously, the flux contains sodium hydroxide.

The sample is preferably heated inductively within a graphite crucible in an induction furnace.

The predetermined reference temperature profile is typically determined by the characteristics and amount of the ore sample and/or the flux.

Advantageously, the sample is supplied with a high level of heat for a predetermined first period of time and then supplied with a lower level of heat for a predetermined second period of time to form the fusion of slag and lead.

The lead is preferably separated from the slag by way of a centrifugal separator.

Advantageously, the lead separated from the slag is poured into a chilled mould, to provide a solid lead button.

Advantageously, the solid lead button is then analysed by way of SAFT analysis.

According to another aspect of the invention there is provided a method as described above including the steps of storing information on each sample on a central database, providing each bottle into which the sample is poured with a unique bar code, scanning each bottle before inserting it into the induction furnace, correlating the bar code and information on the central database, and applying a predetermined reference temperature profile to the sample, according to the information on the sample stored in the database.

Preferably, each lead button is stamped with an identification code.

According to another aspect of the invention there is provided an induction furnace comprising:

a housing formed from ceramic material;

a graphite crucible for containing an ore sample to be melted locatable within the housing;

a barrier, formed from ceramic material, which is arranged to surround the graphite crucible, when it is within the housing;

an inductive coil arranged to surround the barrier; and

a lid which is arranged to engage sealingly with the opening into the graphite crucible.

BRIEF DESCRIPTION OF THE DRAWINGS

The invention will now be described in more detail, by way of example only, with reference to the accompanying drawings in which Figures 1 to 5 illustrate AN embodiment of the invention.

Figure 1	is a schematic flow diagram of a process according to the invention for assaying an ore sample to determine the concentration of selected metals therein;
Figure 2	is a pictorial view of a bottle containing flux;

Figure 3	is a schematic top plan view of an apparatus according to the invention for carrying out the process of Figure 1;
Figure 4	is a cross-sectional view of an induction furnace according to the invention; and
Figure 5	is a graph of electrical energy versus time, showing a typical energy profile for heating the induction furnace of Figure 4; and

DESCRIPTION OF AN EMBODIMENT

Referring to Figure 1, according to the invention there is provided a method of assaying an ore sample, the method including the steps of preparing an ore sample 10, mixing the ore sample 10 with a flux 12 which includes lead oxide, to form a mixture 14, inductively heating the mixture 14 to form of fusion of slag 16 and lead 18 containing gold and PGMs, separating the lead 18 from the slag 16 and determining the amount of gold and PGMs in the lead.

In the sample preparation step, an ore sample is logged and its bulk weight determined. The ore sample is then dried, comminuted and sieved and split at into a number of aliquots which are deposited into storage containers in the form of small plastic bottles, each of which already contains a flux. Information on the sample, such as weight, type etc is entered onto a computer and a bar code label is produced for each sample. The unused portion of the ore sample is sealed in a storage container and identified by means of a bar code label indicating identification, origin and date of assay.

A typical bottle 20 into which an ore sample is placed is shown in Figure 2. The bottle 20 is made from a plastics material (polyethylene) and has a lid 22 which engages sealingly with the opening into the bottle 20. A charge of flux 26 is shown in the bottle 20. Each bottle 20 is identified by means of a printed bar code label 28 indicative of sample identification and weight, which is attached to the top surface of the lid 22. The bottle is also provided with a ridge 30 just below the lid 22.

After a sample has been added to the bottle, the lid is applied back onto the bottle and the sample and flux is mixed by merely shaking the bottle. Previously, mixing containers were used which caused spillages and contamination between samples.

It is envisaged that the sample preparation step is automated as a series of mechanically linked steps, enabling the preparation of an ore sample for assaying without the need for manual intervention.

The flux 26 contains lead oxide, sodium hydroxide, borax and may also contain a small amount of silver nitrate. It is important that the lid 22 engages sealingly with the opening 24 to the bottle 20 to ensure that the sodium hydroxide (NaOH) which is corrosive and hygroscopic does not come into contact with the atmosphere. The flux 26 is specially prepared for use in an induction furnace and is described in detail in a co-pending provisional patent application entitled "assaying fluxes" filed in the name of the applicant of this application, on the same date as this application and which is incorporated herein by reference. The constitution of the flux is determined by the characteristics of the ore which is to be tested, in particular, the amount of silicates and the concentration of gold and PGMs in the ore.

Referring to Figure 3, bottles 20 which contain sample mixed with flux are loaded onto a sample loader 34 which comprises a conveyor belt 36, an optical sensor 38, a bar code reader 40, and a mechanical gripper arm 42. In use, the optical sensor 38 senses the presence of a bottle 20, the bar code reader 40 (positioned above the bottle, and shown in dotted outline) scans the bar code 28 on the lid of the bottle 20 and the gripper arm 42 grips the bottle at the ridge 30 and transfers it into an induction furnace 44 which is powered by a generator 46. The conveyor 36 conveys the next bottle 20 in line with the sensor 38 and this next bottle is then ready to be transferred into the induction furnace 44.

Referring to Figure 4, the induction furnace 44 comprises a removable graphite crucible 46 which is made from soft grain graphite. The crucible 46 is surrounded by a barrier 47 of ceramic material (1600 heat isolating) which in turn is surrounded by an induction coil 48. The abovementioned components are housed in a housing 50 which is made from a ceramic material (polyester board GPO3). The furnace 44 also has a lid 52 which has a backing 54 also made from the ceramic material and an underside 56, made from alumina based ceramic material, which engages sealingly with the opening 58 into the crucible 46, when the lid 52 is closed. It is important that the lid 52 seals the crucible 46, to create a reducing atmosphere therein, so that the graphite is not oxidised at high temperatures within the furnace.

Once a bottle 20 has been inserted into the graphite crucible 46 the lid 52 is closed, the bottle 20 and mixture of sample and flux therein are heated according to a predetermined temperature profile, which is selected according to the amount and characteristics of ore with is being assayed and also the amount and characteristics of the flux 26. Generally, the coil 48 is supplied with 50 kW of electrical energy from the generator 46 for a predetermined first period of time (generally 20 to 30 seconds) and then dropped to 20 to 30kW for

a predetermined second period of time (generally 20 to 30 seconds). Thus, fusion of the ore and flux takes approximately 60 seconds.

Figure 5 shows a typical temperature profile of electrical energy versus time for a heating process according to the invention.

Fusion of the ore and flux is extremely quick and the alternating electro-magnetic waves (eddy waves) from the induction coil causes a violent stirring action of molten lead within the crucible which results in complete collection of gold and PGMs by the lead within about 60 seconds of power being applied to the induction furnace.

Furthermore, the combination of graphite crucible 46 and ceramic housing 50 ensures that, while the temperature within the crucible 46 can reach up to 1500°C, the outer surface of the ceramic housing 50 does not reach more that 60 to 100°C. This is because the ceramic housing 50 is not heated by eddy waves from the induction coil 48. The ceramic barrier 47 (which is not heated by the induction coil) serves to insulate the crucible 46 so that radiant energy is isolated within the crucible.

The plastics bottle 20 is an important part of the heating process as it provides a carbon source for reducing the lead oxide to produce molten lead. The graphite crucible also provides a source of carbon.

Another important aspect of the invention is that the temperature profile applied to each sample can be computer-controlled and thus the heating conditions can be accurately controlled, according to the identification made by the bar code reader. Information on each sample, such as sample type, sample weight, flux

weight, flux composition, coppering pattern, fusion conditions (ie. temperature profile) can be processed through and by the system and this inforamtion used to control the temperature profile. The system can also be used to subject similar ores, or ores from the same area, to similar or the same conditions, to ensure consistent assay results.

Referring back to Figure 3, after the sample has been heated in the induction furnace 44, the molten sample, which comprises lead and slag, is poured into a centrifugal slag separator 60 which is described in a co-pending provisional patent application entitled "slag separator" filed in the name of the applicant of this application, on the dame date as this application and which is incorporated herein by reference. After the slag and lead have been separated, molten lead is cooled rapidly, by chilling the lead. Rapid cooling stops gold and PGMs in the lead from forming layers ensuring that the concentration of gold and PGMs within the button is homogenous.

Thereafter, the button is stamped with an identification number which corresponds to the bar code which was read at the beginning of the process and the buttons are stored on a button storage rack 62.

The amount of gold and PGMs within the button may be analysed by any conventional process, for example by cupellation, which takes approximately 60 minutes. The main advantage of homogenous button 74 produced by the process of the invention is that it can be analysed by way of SAFT analysis. In prior art processes, due to the separation and cooling techniques employed, PGMs and gold form layers within the lead and the lead cannot be analysed by SAFT. As SAFT analysis only takes approximately 30 seconds, the use of SAFT analysis on the lead buttons produced by the invention dramatically reduces the time taken up by analysis. Thus, when the process according to the invention is used and the homogenous button is analysed by way of SAFT

analysis, the whole analysis process could be done in far less time than the twelve hour turn around time of known processes.

DATED THIS 29TH DAY OF OCTOBER 1998.

SPOOR AND FISHER
APPLICANTS PATENT ATTORNEYS

FIG 1

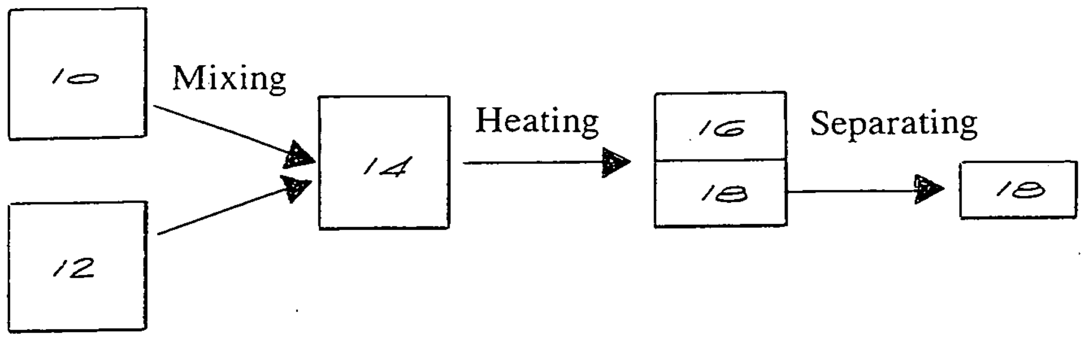


FIG 2

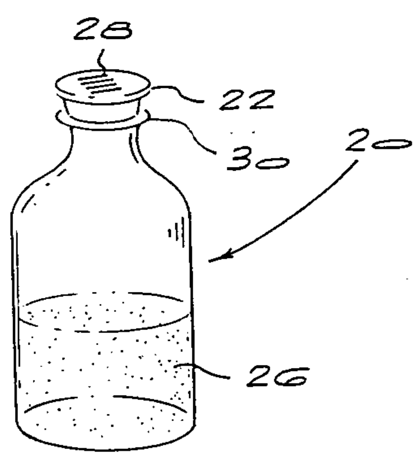
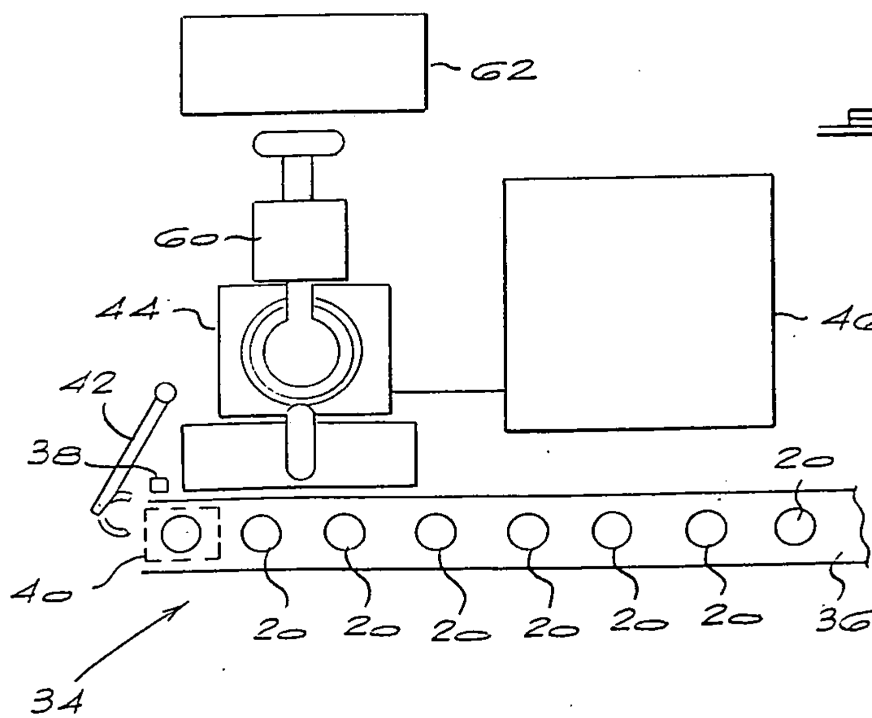


FIG 3



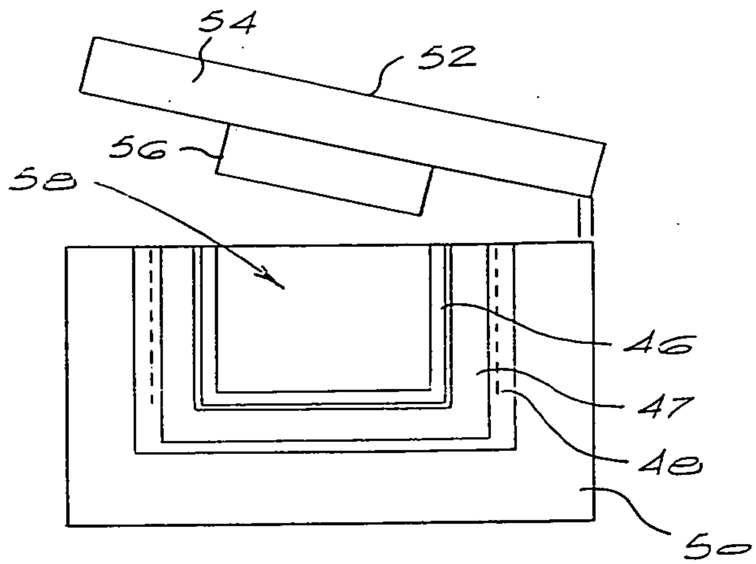


FIG 4

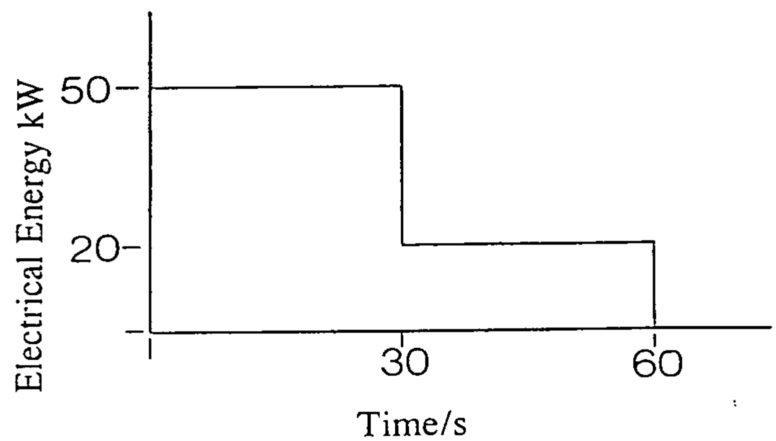


FIG 5